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on July 27, 2001

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Signature

PATENT

#Y2-0281-UNI
Case #F3266(C)

176#
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88-70-01
CW

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Blyth et al.
Serial No.: 09/854,877
Filed: May 14, 2001
For: AMBIENT STABLE BEVERAGE

Edgewater, New Jersey 07020
July 27, 2001

SUBMISSION OF PRIORITY DOCUMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

Pursuant to rule 55(b) of the Rules of Practice in Patent Cases, Applicant(s) is/are submitting herewith a certified copy of the United Kingdom Application No. 0011676.4 filed May 15, 2000, upon which the claim for priority under 35 U.S.C. § 119 was made in the United States.

It is respectfully requested that the priority document be made part of the file history.

Respectfully submitted,

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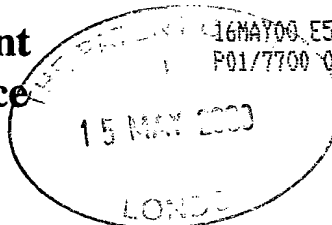
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Request for grant of a patent

(See the notes on the back of this form. You can also get an explanatory leaflet from the Patent Office to help you fill in this form)

1.	Your reference	F3266 (C)/sje		
2.	Patent application number (The Patent Office will fill in this part)	15 MAY 2000	0011676.4	
3.	Full name, address and postcode of the or of each applicant (underline all surnames)	UNILEVER PLC UNILEVER HOUSE, BLACKFRIARS LONDON, EC4P 4BQ		
	Patents ADP number (if you know it)	00001628002		
	If the applicant is a corporate body, give the country/state of its incorporation	UNITED KINGDOM		
4.	Title of the invention	AMBIENT STABLE BEVERAGE		
5.	Name of your agent (if you have one)	EVANS, Jacqueline Gail		
	"Address for Service" in the United Kingdom to which all correspondence should be sent (including the postcode)	PATENT DEPARTMENT, UNILEVER PLC COLWORTH HOUSE, SHARNBROOK BEDFORD, MK44 1LQ		
	Patents ADP number (if you know it)	07367642001		
6.	If you are declaring priority from one or more earlier patent applications, give the country and the date of filing of the or of each of these earlier applications and (if you know it) the or each application number	Country	Priority application number (if you know it)	Date of filing (day / month / year)
7.	If this application is divided or otherwise derived from an earlier UK application, give the number and the filing date of the earlier application	Number of earlier application		Date of filing (day/month/year)
8.	Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if: a) any applicant named in part 3 is not an inventor, or b) there is an inventor who is not named as an applicant, or c) any named applicant is a corporate body. See note (d))	YES		

Patents Form 1/77

9. Enter the number of sheets for any of the following items you are filing with this form. Do not count copies of the same document

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Description	10
Claim(s)	2
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Priority Documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (*Patents Form 7/77*)

Request for preliminary examination and search (*Patents Form 9/77*) 1

Request for substantive examination (*Patents Form 10/77*)

Any other documents
(please specify)

11. I/We request the grant of a patent on the basis of this application.

Signature(s)  Date: 15-May-00

Sandra Jane EDWARDS, Authorised Signatory

12. Name and daytime telephone number of person to contact in the United Kingdom Sandra Edwards, Tel 01234 22 2068

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AMBIENT STABLE BEVERAGE5 **Field of the invention**

The present invention relates to an ambient stable beverage, particularly a tea based beverage, that is preserved by a minimal amount of sorbic acid.

10

Background and prior art

In recent years there has been an ever increasing choice for consumers who wish to quench their thirst with ready made beverages. Many of those are now turning from the well known soft drinks to tea based beverages, be those carbonated or still, and the "natural" refreshment they can provide.

20 Tea contains a complex combination of enzymes, biochemical intermediates and structural elements normally associated with plant growth and photosynthesis. There are also many natural substances that give tea its unique taste, astringency, aroma and colour. Many of these are produced by the oxidation reactions
25 that occur during the so-called fermentation stage of black tea manufacture. Tea production has long been driven by traditional processing methods with only a fundamental understanding of the chemistry that is involved. As a consequence manufacturers have discovered making ambient stable tea based beverages at the
30 volumes required to compete with more traditional soft drinks is not simply a matter of flavouring a soft drink with tea.

The flavour of a tea based beverage and its stability rely on the stability of the beverage as a whole. The fungi including yeasts
35 and moulds that can grow in tea based beverages and other soft

- 2 -

drinks can be killed by heat treatment or at least controlled by use of preservatives. Some tea based beverages are therefore pasteurised and then bottled in glass or special heat stable PET containers. This is known as "hot filling". Unfortunately this
5 can be an expensive operation that creates a great deal of environmentally unfriendly waste. It has therefore become more attractive for manufacturers to pack their tea based products in standard PET containers which can range from single serve units to multi-serve packs and maintain the stability of the product using
10 tailor made flavour and preservative systems. This is known as "cold filling". It is also useful in that one can readily use a tea concentrate or powder.

Potassium sorbate is well known preservative. It is a mould and
15 yeast inhibitor and one of the few legally permitted preservatives of soft drinks and fruit juices. It has been listed in the UK Preservatives in Food regulations since at least 1962. The levels of use tend to be in the range of 100-1000 ppm. That has been found to be an effective antimicrobial agent in a variety of foods
20 including carbonated beverages in certain fruit and vegetable products, including wines. It is sorbic acid that is the effective agent.

Unfortunately even moderate levels of sorbic acid can seriously
25 affect the flavour of a tea based beverage. Adding a strong flavour such as lemon can offset the preservative taste. However consumers are keen to experience other flavours, often more delicate flavours. Furthermore, some of those consumers that were drawn to tea based products as a more healthy and natural
30 alternative to soft drinks would reduce their intake of preservatives generally.

Accordingly there is a need for pleasantly flavoured, ambient-stable, tea based beverages that contain minimal amounts of
35 preservatives such as sorbic acid. Non-tea based beverages

including fruit and soft drinks can be stabilised in a similar way.

5 In response to that need the present inventors have now developed an ambient stable beverage that is preserved by a minimal amount of sorbic acid.

Statement of the Invention

10

The invention can in broad terms be said to relate to an ambient stable beverage, particularly a tea based beverage, that contains a preservative system comprising 10 to 200 ppm sorbic acid and at least one essential oil. When the beverage is tea based it
15 preferably contains 0.01 to 3% tea solids, especially about 0.14% tea solids.

The beverage preferably contains 1 to 100 ppm of the essential oil, except when the essential oil is cinnamic the beverage
20 preferably contains 1 to 175 ppm cinnamic acid.

The invention can also be said to relate to a method for preparing an ambient-stable tea based beverage suitable for cold filing comprising preserving a tea extract with a preservative system
25 comprising 10 to 200 ppm sorbic acid and an essential oil.

"Beverage" for the purposes of the present invention means any drink, other than water, and includes soft drinks, fruit drinks, coffee based drinks and tea based drinks.

30

"Essential oil" for the purposes of the present invention includes any of the volatile oils in plants having the odour or flavour of the plant from which they are extracted. It also includes one or more of the components of that oil that is or are responsible for
35 or at least contributes to the odour or flavour of that plant.

"Tea" for the purposes of the present invention means leaf material from *Camellia sinensis* var. *sinensis* or *Camellia sinensis* var. *assamica*. "Tea" is also intended to include the product of blending two or more of any of these teas.

5

For the avoidance of doubt the word "comprising" is intended to mean including but not necessarily "consisting of" or "composed of". In other words the listed steps or options need not be exhaustive.

10

Except in the operating and comparative examples, or where otherwise explicitly indicated, all numbers in this description indicating amounts or concentrations of material ought to be understood as modified by the word "about".

15

Detailed description of the invention

The ambient stable beverage of the present invention contains a preservative system a minimal amount of sorbic acid and an essential oil. The beverage is preferably a tea based beverage but non-tea based beverages including fruit and soft drinks can be stabilised using the same preservative system.

When the beverage is a tea based beverage it will contain a tea extract. The tea extract can be obtained by any suitable means. Preferably tea leaves are extracted in hot water over a period of between 20 minutes and 5 hours. The extract can be dried to form a powder, reconstituted to form an acidic beverage, or concentrated to form a syrup from which one can prepare a tea based beverage.

Tea is known to have certain antibacterial and antiviral properties in itself. One must exceed a concentration of about 3% to evidence tea beginning to suppress the growth of yeasts and

moulds. At concentrations lower than this, which is typical for tea based beverages, tea acts as a nutrient that enhances the potential for microbial spoilage. The beverage should therefore contain 0.01 to 3% tea solids, about 0.14% being particularly preferred.

The preservative system comprises 10 to 200 ppm sorbic acid and an essential oil.

10 The inventors tested the following compounds: acetaldehyde, 2-acetylfuran, amyl acetate, amyl alcohol, α -amylcinnamaldehyde, amyl formate, trans-anethole, m-anisaldehyde, o-anisaldehyde, p-anisaldehyde, anisole, anisyl alcohol, benzaldehyde, benzaldehyde dimethyl acetal, benzoin, benzophenone, 15 benzothiazole, benzyl acetate, benzyl acetoacetate, benzyl alcohol, benzyl benzoate, benzyl cinnamate, benzyl ether (dibenzyl ether), benzyl formate, benzyl-4-hydroxybenzoate, biphenyl, borneol, butanal, 1-butanol, 2-butanone, butyl acetate, tert-butyl acetoacetate, butyl butyrate, 4-tert-butylcyclohexanone, tert- 20 butyl ethyl malonate, butyl formate, butyl lactate, butyl levulinate, butyl phenyl ether, butyl propionate, butyric acid, γ -butyrolactone, caffeic acid, caffeine, (+)-camphene, (-)-camphene, campher, carvacrol, carveol, carvone, carvyl acetate, carvyl propionate, caryophyllene oxide, cedarwood oil, cineole, 25 cinnamaldehyde, cinnamyl acetate, cinnamyl alcohol, cinnamyl chloride, cinnamyl formate, trans-cinnamic acid, cinnamon oil, trans-cinnamoyl chloride, citral, citral dimethyl acetal, (S)-citronellic acid, (R)-citronellic acid, citronellal, citronellol, coumaric acid, creosol, m-cresol, o-cresol, p-cresol, cumene, 30 cumic acid, cumic alcohol, cuminaldehyde, cumic aniline, cyclohexanebutyric acid, cyclohexyl acetate, cyclohexylacetic acid, 2-cyclohexylethyl acetate, p-cymene, trans,trans-2,4-decadienal, decanal, decanol, δ -decanolactone, 3-decanone, decanoic acid, trans-4-decenal, diacetyl (2,3-butanedione), 35 diethyl malonate, 2,3-diethyl pyrazine, diethyl succinate, diethyl

L-tartrate, dihydrocarveol, dihydrocarvone, dihydrocoumarin, 2,6-dimethyl-4-heptanol, 2,6-dimethyl-5-heptenal (melonal), 3,7-dimethyl-1-octanol, 2,3-Dimethyl pyrazine, dimethyl succinate (DBE-4), dodecane, estragole (4-allylanisole), ethyl acetate, ethyl butyrate, ethyl cyclohexanepropionate, ethyl decanoate (caprate), ethyl formate, ethyl heptanoate, ethyl hexanoate, 2-ethyl-1-hexanol, ethyl myristate, ethyl nonanoate, ethyl octanoate (caprylate), ethyl palmitate, ethyl propionate, ethyl pyruvate, ethyl sorbate, ethyl tridecanoate, ethyl undecanoate, ethyl valerate, ethyl vanillin, eugenol, ferulic acid, fumaric acid, geranic acid, geraniol, geranyl acetate, glyceryl tribenzoate (tribenzoin), glycyrrhizic acid, guaiacol, heptanal, heptanoic acid, 1-heptanol, hexanal, hexanoic acid (caproic), 1-hexanol, 2-hexanol, 3-hexanol, 3-hexanone, trans-2-hexenoic acid, trans-3-hexenoic acid, cis-2-hexen-1-ol, trans-2-hexen-1-ol, hexyl acetate, 4-hexylbenzoic acid, trans- β -hydromuconic acid, m-hydroxybenzoic acid, p-hydroxybenzoic acid, o-hydroxybiphenyl, hydroxycitronellal, γ -ionone, isoamyl acetate, isobutyl acetate, isobutyric acid, isoeugenol, isopropyl acetate, jasmone, leucine, limonene, linalool, linalyl acetate, menthol, menthone, 4-methoxybenzyl alcohol, o-methoxycinnamaldehyde, 4-(p-methoxyphenyl)-2-butanone, methyl acetate, methyl anthranilate, methyl butyrate, α -methyl-trans-cinnamaldehyde, methyl decanoate, methyl eugenol, methyl heptanoate (enantate), methyl hexanoate (caproate), methyl laurate, methyl myristate, methyl nonanoate, methyl octanoate (caprylate), 2-methyl-2-pentenal, 5-methyl-2-phenyl-2-hexenal, methyl propionate, methyl salicylate, 4-methyl-5-thiazole ethanol, 4-methyl-5-thiazoleethanol acetate, methyl tridecanoate, methyl valerate, methyl undecanoate, β -myrcene, 7-methyl-3-methylene-1,6-octadiene, myristaldehyde, myrtenol, neomenthol, nerol, nerolidol, nonanal, nonanoic acid, γ -nonanoic lactone, 1-nonanol, δ -octalactone, octanal, octanoic acid (caprylic), 1-octanol, octyl acetate, pentanal, pentanol, phenylacetic acid, phenylacetone, 1-phenyl-1,2-propanedione, 2-phenylpropionic acid, 3-phenylpropionic acid (hydrocinnamic acid),

pinene, piperonyl acetate, propanal, 1-propanol, 2-propanol
 (isopropanol), propenylguaethol, propyl acetate, propyl benzoate,
 pulegone, quinine hydrochloride, safrole, salicylaldehyde, skatole
 (3-methylindole), sorbic alcohol (2,4-hexandienol), sorbic
 5 aldehyde (2,4-hexadienal), tartaric acid, α -terpinene, γ -
 terpinene, terpinen-4-ol, terpineol, tolualdehyde, thymol,
 triacetin (glyceryl triacetate), tributyl acetylcitrate,
 tributyrin, 3,5,5-trimethyl-1-hexanol, γ -undecalactone, undecanal,
 undecane, undecanoic acid, 1-undecanol, 2-undecanol, valeric acid,
 10 vanillic acid, vanillin, vanillyl alcohol and veratraldehyde.

Table 1 below contains those of essential oils listed above that
 exhibited a fungicidal activity suitable for use in the present
 invention. The minimum inhibitory concentration (MIC) is given
 15 for each compound.

TABLE I
 Preferred essential oils

	<u>COMPOUND</u>	<u>MIC (ppm)</u>
	Benzyl-4-hydroxybenzoate	68
	4-tert-Butylcyclohexanone	462
25	Carvone	300
	Cinnamaldehyde	66
	Cinnamic acid	148
	Citral	228
	Citral dimethyl acetal	198
30	Citronellol	125
	Cumic alcohol	450
	Cyclohexanebutyric acid	68
	2-Cyclohexylethyl acetate	102
	trans,trans-2,4-Decadienal	8
35	Decanal	47
	Decanol	24
	Dihydrocarveol	540
	3,7-Dimethyl-1-octanol	15.8
	Ethyl cyclohexanepropionate	184
40	Ethyl pyruvate	1392
	Ethyl vanillin	249
	Jasmone	246
	o-Methoxycinnamaldehyde	130

	Methyl anthranilate	310
	α -Methyl-trans-cinnamaldehyde	58.4
	Methyl eugenol	356
	Methyl nonanoate	90
5	2-Methyl-2-pentenal	1274
	5-Methyl-2-phenyl-2-hexenal	162
	Methyl salicylate	152
	4-Methyl-5-thiazoleethanol acetate	1110
	Myrtenol	137
10	Neomenthol	156
	Nonanoic acid	63
	γ -Nonanoic lactone	63
	δ -Octalactone	568
	Octanoic acid (caprylic)	115
15	1-Octanol	247
	1-Phenyl-1,2-propanedione	222
	Piperonyl acetate	242
	Propyl benzoate	66
	Pulegone	152
20	Sorbic aldehyde (2,4-hexadienal)	86
	Terpinen-4-ol	616
	Tolualdehyde	240
	γ -Undecalactone	28
	Undecanal	34
25	1-Undecanol	14
	Vanillin	1216

- 30 The preservative system preferably contains 1 to 100 ppm of the essential oil, except when the essential oil is cinnamic acid the system can contain 1 to 175 ppm cinnamic acid.

- 35 Some of the aforementioned essential oils were found to be particularly preferred in respect of their impact on the taste profile of tea based beverages containing them. These are listed in Table II below. In each case the respective minimum inhibitory concentration (MIC) and preferred concentration is also given.

TABLE II
Particularly preferred essential oils

	<u>COMPOUND</u>	<u>MIC (ppm)</u>	<u>Conc (ppm)</u>
5	Cinnamic acid	148	1-60
	Citral	228	1-30
	Citral dimethyl acetal	198	1-30
	Cumic alcohol	450	1-40
10	trans,trans-2,4-Decadienal	8	1-20
	3,7-Dimethyl-1-octanol	15.8	1-20
	Ethyl pyruvate	1392	1-40
	Myrtenol	137	1-20
	Piperonyl acetate	242	1-20

15

An especially preferred preservative system for tea based beverages, based on preservative action and taste profile comprises 1 to 30 ppm cinnamic acid, 1 to 30 ppm citral dimethyl acetal, 1 to 40 ppm cumic alcohol (isopropylbenzyl alcohol), and 1 to 20 myrtenol and piperonyl acetate.

25

Water quality can seriously undermine the stability of a beverage. This is a particularly important factor when making a tea based beverage for cold filing. For that purpose it will often be important to minimise the yeast content of water used at all stages of production. Art known methods include chlorination/dechlorination and UV irradiation.

30

Ambient-stable beverages of the invention may be still or carbonated. Carbonation appears to provide a preservative effect in itself and therefore the formulation of a carbonated product need not be the same as a still one.

35

Tea based beverages commonly contain sugar or some other sweetener to counter the sometimes astringent taste of tea. Most microbes that can typically grow in tea based beverages thrive on sugar, a source of nitrogen, oxygen, zinc, magnesium, potassium, phosphate and vitamins. It is therefore advantageous to limit the sugar

content to 8 to 10 degrees brix, however one could use up to 60 degrees brix when the product is a tea mix.

Oxygen content can be minimised by pre-pasteurisation or some heat treatment or nitrogen sparging. The mineral content of a tea based beverage can be minimised using EDTA, citrate, or a water softener. For example microbes can grow in tea if the concentration of magnesium ions exceeds 0.2 ppm, and they only need trace levels of zinc.

The present invention also relates to a method for preparing a method for preparing an ambient-stable tea based beverage that is suitable for cold filing. The method comprises preserving a tea extract with a preservative system comprising 10 to 200 ppm sorbic acid and one or more of the aforesaid essential oils.

CLAIMS

1. A beverage that contains a preservative system comprising 10 to 200 ppm sorbic acid and at least one essential oil.
2. A beverage according to claim 1 wherein the essential oil is selected from the group consisting of benzyl-4-hydroxybenzoate, 4-tert-butylcyclohexanone, carvone, cinnamaldehyde, cinnamic acid, citral, citral dimethyl acetal, citronellol, cumic alcohol, cyclohexanecarboxylic acid, 2-cyclohexylethyl acetate, trans,trans-2,4-decadienal, decanal, decanol, dihydrocarveol, 3,7-dimethyl-1-octanol, ethyl cyclohexanecarboxylate, ethyl pyruvate, ethyl vanillin, jasmone, o-methoxycinnamaldehyde, methyl anthranilate, α -methyl-trans-cinnamaldehyde, methyl eugenol, methyl nonanoate, 2-methyl-2-pentenal, 5-methyl-2-phenyl-2-hexenal, methyl salicylate, 4-methyl-5-thiazoleethanol acetate, myrtenol, neomenthol, nonanoic acid, γ -nonanoic lactone, δ -octalactone, octanoic acid (caprylic), 1-octanol, 1-phenyl-1,2-propanedione, piperonyl acetate, propyl benzoate, pulegone, sorbic aldehyde (2,4-hexadienal), terpinen-4-ol, tolualdehyde, γ -undecalactone, undecanal, 1-undecanol, and vanillin.
3. A beverage according to claim 2 wherein the essential oil is selected from the group consisting of cinnamic acid, citral, citral dimethyl acetal, cumic alcohol (isopropylbenzyl alcohol), trans,trans-2,4-decadienal, 3,7-dimethyl-1-octanol, ethyl pyruvate, myrtenol and piperonyl acetate.
4. A beverage according to any preceding claim wherein the preservative system contains 50 to 150 ppm sorbic acid.

5. A beverage according to any preceding claim wherein the preservative system contains 1 to 100 ppm of one or more of the essential oils.
- 5 6. A beverage according to any claim 1 or 4 wherein the preservative system contains 1 to 175 ppm cinnamic acid as the essential oil.
7. A beverage according to any preceding claim wherein the
10 beverage is a tea based beverage.
8. A beverage according to claim 7 wherein the beverage contains 0.01 to 3% tea solids.
- 15 9. A method for preparing an ambient-stable tea based beverage suitable for cold filing comprising preserving a tea extract with a preservative system comprising 10 to 200 ppm sorbic acid and an essential oil.

ABSTRACT

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Ambient stable beverage

A beverage that contains a preservative system that contains 10 to 200 ppm sorbic acid and one or more essential oils.

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